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Docket No. VIT 0012 PA

METHOD FOR EDGE SEALING BARRIER FILMS

Background of the Invention

The invention relates generally to multilayer, thin film barrier composites, and more particularly, to multilayer, thin film barrier composites having the edges sealed against lateral moisture and gas diffusion.

Multilayer, thin film barrier composites having alternating layers of barrier material and polymer material are known. These composites are typically formed by depositing alternating layers of barrier material and polymer material, such as by vapor deposition. If the polymer layers are deposited over the entire surface of the substrate, then the edges of the polymer layers are exposed to oxygen, moisture, and other contaminants. This potentially allows the moisture, oxygen, or other contaminants to diffuse laterally into an encapsulated environmentally sensitive device from the edge of the composite, as shown in Fig. 1. The multilayer, thin film barrier composite 100 includes a substrate 105 and alternating layers of decoupling material 110 and barrier material 115. The scale of Fig. 1 is greatly expanded in the vertical direction. The area of the substrate 105 will typically vary from a few square centimeters to several square meters. The barrier layers 115 are typically a few hundred Angstroms thick, while the decoupling layers 110 are generally less than ten microns thick. The lateral diffusion rate of moisture and oxygen is finite, and this will eventually compromise the encapsulation. One way to reduce the problem of edge diffusion is to provide long edge diffusion paths. However, this decreases the area of the substrate which is usable for active environmentally sensitive devices. In addition, it only lessens the problem, but does not eliminate it.

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A similar edge diffusion problem will arise when a substrate containing a multilayer, thin film barrier composite is scribed and separated to create individual components.

Thus, there is a need for a edge-sealed barrier film composite, and for a method of making such a composite.

Summary of the Invention

The present invention solves this need by providing an edge-sealed barrier film composite. The composite comprises a substrate, and at least one initial barrier stack adjacent to the substrate, the at least one initial barrier stack comprising at least one decoupling layer and at least one barrier layer, wherein a first decoupling layer has an area and wherein a first barrier layer has an area, the area of the first barrier layer being greater than the area of the first decoupling layer, and wherein the first decoupling layer is sealed by the first barrier layer within the area of the first barrier layer. By adjacent, we mean next to, but not necessarily directly next to. There can be additional layers intervening between the substrate and the barrier stacks.

The first layer can be either a decoupling layer or a barrier layer, as can the last layer. One or more barrier stacks can include at least two decoupling layers and/or at least two barrier layers. When a barrier stack has at least two barrier layers, a second barrier layer may have an area greater than the area of the first decoupling layer, and the first and second barrier layers may seal the first decoupling layer between them.

The decoupling layers can be made from materials including, but not limited to, organic polymers, inorganic polymers, organometallic polymers, hybrid organic/inorganic polymer systems, silicates, and combinations thereof. The decoupling layers can be made of the same

decoupling material or different decoupling materials.

Suitable barrier materials include, but are not limited to, metals, metal oxides, metal nitrides, metal carbides, metal oxynitrides, metal oxyborides, and combinations thereof. Suitable barrier materials also include, but are not limited to, opaque metals, opaque ceramics, opaque polymers, and opaque cermets, and combinations thereof. The barrier layers can be made of the same barrier material or different barrier material.

The composite can include an environmentally sensitive device. Environmentally sensitive devices include, but are not limited to, organic light emitting devices, liquid crystal displays, displays using electrophoretic inks, light emitting diodes, light emitting polymers, electroluminescent devices, phosphorescent devices, electrophoretic inks, organic solar cells, inorganic solar cells, thin film batteries, and thin film devices with vias, and combinations thereof.

Another aspect of the invention is an edge-sealed, encapsulated environmentally sensitive device. The edge-sealed, encapsulated environmentally sensitive device includes: at least one initial barrier stack comprising at least one decoupling layer and at least one barrier layer, wherein a first decoupling layer of a first initial barrier stack has an area and wherein a first barrier layer of the first initial barrier stack has an area, the area of the first barrier layer of the first initial barrier stack being greater than the area of the first decoupling layer of the first initial barrier stack, and wherein the first decoupling layer of the first initial barrier stack is sealed by the first barrier layer of the first initial barrier stack within the area of the first barrier layer; an environmentally sensitive device adjacent to the at least one initial barrier stack; and at least one

additional barrier stack adjacent to the environmentally sensitive device on a side opposite the at least one initial barrier stack, the at least one additional barrier stack comprising at least one decoupling layer and at least one barrier layer, wherein a first decoupling layer of a first additional barrier stack has an area and wherein a first barrier layer of the first additional barrier stack has an area, the area of the first barrier layer of the first additional barrier stack being greater than the area of the first decoupling layer of the first additional barrier stack, wherein the first decoupling layer of the first additional barrier stack is sealed by the first barrier layer of the first additional barrier stack within the area of the first barrier layer, and wherein the environmentally sensitive device is sealed between the at least one initial barrier stack and the at least one additional barrier stack.

Another aspect of the invention is a method of making an edge-sealed barrier film composite. The method includes providing a substrate, and placing at least one initial barrier stack adjacent to the substrate, the at least one initial barrier stack comprising at least one decoupling layer and at least one barrier layer, wherein a first decoupling layer of a first initial barrier stack has an area and wherein a first barrier layer of the first initial barrier stack has an area, the area of the first barrier layer being greater than the area of the first decoupling layer, and wherein the first decoupling layer is sealed by the first barrier layer within the area of the first barrier layer.

Placing the at least one barrier stack adjacent to the substrate includes, but is not limited to, depositing the at least one barrier stack adjacent to the substrate, and laminating the at least one barrier stack adjacent to the substrate.

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The barrier layers can be deposited before or after the decoupling layers, depending on the particular application and structure.

Depositing the at least one barrier stack may include, but is not limited to, providing a mask with at least one opening, depositing the first decoupling layer through the at least one opening in the mask, and depositing the first barrier layer.

Alternatively, depositing the at least one barrier stack adjacent to the substrate may include, but is not limited to, depositing the first decoupling layer having an initial area of decoupling material which is greater than the area of the first decoupling layer, etching the first decoupling layer having the initial area of decoupling material to remove a portion of the decoupling material so that the first decoupling layer has the area of the first decoupling layer, and depositing the first barrier layer. Etching the first decoupling layer may include, but is not limited to, providing a solid mask over the first decoupling layer having the initial area of decoupling material, and etching the first decoupling layer having the initial area of decoupling material to remove the portion of the decoupling material outside the solid mask so that the first decoupling layer has the area of the first decoupling layer. The first decoupling layer may be etched so that at least one edge of the first decoupling layer has a gradual slope. A reactive plasma may be used to etch the decoupling layers. Reactive plasmas include, but are not limited to O_2, CF_4, H_2, and combinations thereof.

The method may include placing an environmentally sensitive device adjacent to the substrate before the at least one initial barrier stack is placed thereon. Alternatively, the method may include placing the environmentally sensitive device adjacent to the at least one initial

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barrier stack after the at least one initial barrier stack is placed on the substrate. The method may also include placing at least one additional barrier stack adjacent to the environmentally sensitive device on a side opposite the substrate, the at least one additional barrier stack comprising at least one decoupling layer and at least one barrier layer, wherein a first decoupling layer of a first additional barrier stack has an area and wherein a first barrier layer of the first additional barrier stack has an area, the area of the first barrier layer of the first additional barrier stack being greater than the area of the first decoupling layer of the first additional barrier stack, and wherein the first decoupling layer of the first additional barrier stack is sealed by the first barrier layer of the first additional barrier stack within the area of the first barrier layer.

Laminating the at least one barrier stack adjacent to the substrate may be performed using a number of processes including, but not limited to, heat, solder, adhesive, ultrasonic welding, and pressure.

The method may include depositing a ridge on the substrate before depositing the at least one barrier stack adjacent to the substrate, the ridge interfering with the deposition of the first decoupling layer so that the area of the first barrier layer is greater than the area of the first decoupling layer and the first decoupling layer is sealed by the first barrier layer within the area of the first barrier layer.

Brief Description of the Drawings

Fig. 1 is a cross-section of a barrier composite of the prior art.

Fig. 2 is a cross-section of one embodiment of an edge-sealed, barrier composite of the present invention.

Fig. 3 is a cross-section of an embodiment of an edge-sealed, encapsulated environmentally sensitive device of the present invention.

Fig. 4 is a cross-section of a second embodiment of an edge-sealed, encapsulated environmentally sensitive device of the present invention.

Detailed Description of the Invention

Fig. 2 shows one embodiment of an edge-sealed, barrier composite 200. The composite 200 includes a substrate 205. The substrate can be any suitable substrate, and can be either rigid or flexible. Suitable substrates include, but are not limited to: polymers, for example, polyethylene terephthalate (PET), polyethylene naphthalate (PEN), or high temperature polymers, such as polyether sulfone (PES), polyimides, or Transphan™ (a high glass transition temperature cyclic olefin polymer available from Lofo High Tech Film, GMBH of Weil am Rhein, Germany); metals and metal foils; paper; fabric; glass, including thin, flexible, glass sheet (for example, flexible glass sheet available from Corning Inc. under the glass code 0211. This particular thin, flexible glass sheet has a thickness of less than 0.6 mm and will bend at a radium of about 8 inches.); ceramics; semiconductors; silicon; and combinations thereof.

Fig. 2 shows three initial barrier stacks 220 adjacent to the substrate 205. The initial barrier stacks 220 include a decoupling layer 210 and a barrier layer 215. The barrier layer 215 has an area greater than the area of the decoupling layer 210. As a result, the barrier layer 215 extends beyond the edges of the decoupling layer 210, sealing the decoupling layer 210 within the area covered by the barrier layer 215. Because the decoupling layers 210 are sealed within the area covered by the barrier layers 215, ambient moisture, oxygen, and other contaminants

cannot diffuse through the decoupling layers to the environmentally sensitive device.

Fig. 2 shows three initial barrier stacks 220. However, the number of barrier stacks is not limited. The number of barrier stacks needed depends on the substrate material used and the level of permeation resistance needed for the particular application. One or two barrier stacks may provide sufficient barrier properties for some applications. The most stringent applications may require five or more barrier stacks.

Each of the initial barrier stacks 220 shown in Fig. 2 has one barrier layer 215 and one decoupling layer 210. However, the barrier stacks can have one or more decoupling layers and one or more barrier layers. There could be one decoupling layer and one barrier layer, there could be one or more decoupling layers on one side of one or more barrier layers, there could be one or more decoupling layers on both sides of one or more barrier layers, or there could be one or more barrier layers on both sides of one or more decoupling layers. The important feature is that the barrier stack have at least one decoupling layer and at least one barrier layer. The barrier layers in the barrier stacks can be made of the same material or of a different material, as can the decoupling layers. The barrier layers are typically about 100-400 Å thick, and the decoupling layers are typically about 1000-10,000 Å thick.

Although the three initial barrier stacks 220 are shown as having the same layers in the same order, this is not necessary. The barrier stacks can have the same or different layers, and the layers can be in the same or different sequences.

If there is only one barrier stack and it has only one decoupling layer and one barrier layer, then the decoupling layer must be first in order for the barrier layer to seal it, as shown in

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Fig. 2. The decoupling layer will be sealed between the substrate (or the upper layer of the previous barrier stack) and the barrier layer. Although a composite can be made with a single barrier stack having one decoupling layer and one barrier layer, there will typically be at least two barrier stacks, each having one (or more) decoupling layer and one (or more) barrier layer. In this case, the first layer can be either a decoupling layer or a barrier layer, as can the last layer.

Fig. 3 shows an edge-sealed, encapsulated environmentally sensitive device 300. There is a substrate 305 with an environmentally sensitive device 330 adjacent to it. There is a barrier stack 340 adjacent to the environmentally sensitive device 330. The barrier stack includes one decoupling layer 310 and two barrier layers 315, 325. The barrier layer 315 has an area greater than that of the environmentally sensitive device 330. Thus, the environmentally sensitive device 330 is sealed within the barrier layer 315. The barrier layers 315, 325 have an area greater than the area of the decoupling layer 310 so the decoupling layer 310 is sealed between the barrier layers 315, 325.

The environmentally sensitive device can be any device requiring protection from moisture, gas, or other contaminants. Environmentally sensitive devices include, but are not limited to, organic light emitting devices, liquid crystal displays, displays using electrophoretic inks, light emitting diodes, light emitting polymers, electroluminescent devices, phosphorescent devices, electrophoretic inks, organic solar cells, inorganic solar cells, thin film batteries, and thin film devices with vias, and combinations thereof.

It is not required that all of the barrier layers have an area greater than all of the decoupling layers, but at least one of the barrier layers must have an area greater than at least one

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of the decoupling layers. If not all of the barrier layers have an area greater than of the decoupling layers, the barrier layers which do have an area greater than the decoupling layers should form a seal around those which do not so that there are no exposed decoupling layers within the barrier composite, although, clearly it is a matter of degree. The fewer the edge areas of decoupling layers exposed, the less the edge diffusion. If some diffusion is acceptable, then a complete barrier is not required.

Fig. 4 shows an edge-sealed, encapsulated environmentally sensitive device 400. There is a substrate 405 which can be removed after the device is made, if desired. The environmentally sensitive device 430 is encapsulated between two initial barrier stacks 420, 422 on one side and one additional barrier stack 440 on the other side.

Barrier stack 420 has a barrier layer 415 which has an area greater than the area of the decoupling layer 410 which seals the decoupling layer 410 within the area of the barrier layer 415. Barrier stack 422 has two barrier layers 415, 417 and two decoupling layers 410, 412. Barrier layer 415 has an area greater than that of the decoupling layers 410, 412 which seals the decoupling layers 410, 412 within the area of the barrier layer 415. There is a second barrier layer 417.

On the other side of the environmentally sensitive device 430, there is an additional barrier stack 440. Barrier stack 440 includes two decoupling layers 410 and two barrier layers 415 which may be of approximately the same size. Barrier stack 440 also includes barrier layer 435 which has an area greater than the area of the decoupling layers 410 which seals the decoupling layers 410 within the area of barrier layer 435.

The barrier layer which seals the decoupling layer may be the first barrier layer in the barrier stack, as shown in barrier stack 420. It may also be a second (or later) barrier layer as shown in barrier stack 440. Barrier layer 435 which seals the barrier stack 440 is the third barrier layer in the barrier stack following two barrier layers 415 which do not seal the barrier stack. Thus, the use of the terms first decoupling layer and first barrier layer in the claims does not refer to the actual sequence of layers, but to layers which meet the limitations. Similarly, the terms first initial barrier stack and first additional barrier stack do not refer to the actual sequence of the initial and additional barrier stacks.

The barrier stack may include one or more decoupling layers. The decoupling layers may be made from the same decoupling material or different decoupling material. The decoupling layer can be made of any suitable decoupling material, including, but not limited to, organic polymers, inorganic polymers, organometallic polymers, hybrid organic/inorganic polymer systems, silicates, and combinations thereof. Organic polymers include, but are not limited to, urethanes, polyamides, polyimides, polybutylenes, isobutylene isoprene, polyolefins, epoxies, parylenes, benzocyclobutadiene, polynorbornenes, polyarylethers, polycarbonates, alkyds, polyaniline, ethylene vinyl acetate, ethylene acrylic acid, and combinations thereof. Inorganic polymers include, but are not limited to, silicones, polyphosphazenes, polysilazanes, polycarbosilanes, polycarboranes, carborane siloxanes, polysilanes, phosphonitriles, sulfur nitride polymers, siloxanes, and combinations thereof. Organometallic polymers include, but are not limited to, organometallic polymers of main group metals, transition metals, and lanthanide/actinide metals, or combinations thereof. Hybrid organic/inorganic polymer systems

include, but are not limited to, organically modified silicates, preceramic polymers, polyimide-silica hybrids, (meth)acrylate-silica hybrids, polydimethylsiloxane-silica hybrids, ceramers, and combinations thereof.

The barrier stack may include one or more barrier layers. The barrier layers may be made from the same barrier material or different barrier material. The barrier layer can be made from any suitable barrier material. The barrier material can be transparent or opaque depending on what the composite is to be used for. Suitable barrier materials include, but are not limited to, metals, metal oxides, metal nitrides, metal carbides, metal oxynitrides, metal oxyborides, and combinations thereof. Metals include, but are not limited to, aluminum, titanium, indium, tin, tantalum, zirconium, niobium, hafnium, yttrium, nickel, tungsten, chromium, zinc, alloys thereof, and combinations thereof. Metal oxides include, but are not limited to, silicon oxide, aluminum oxide, titanium oxide, indium oxide, tin oxide, indium tin oxide, tantalum oxide, zirconium oxide, niobium oxide, hafnium oxide, yttrium oxide, nickel oxide, tungsten oxide, chromium oxide, zinc oxide, and combinations thereof. Metal nitrides include, but are not limited to, aluminum nitride, silicon nitride, boron nitride, germanium nitride, chromium nitride, nickel nitride, and combinations thereof. Metal carbides include, but are not limited to, boron carbide, tungsten carbide, silicon carbide, and combinations thereof. Metal oxynitrides include, but are not limited to, aluminum oxynitride, silicon oxynitride, boron oxynitride, and combinations thereof. Metal oxyborides include, but are limited to, zirconium oxyboride, titanium oxyboride, and combinations thereof. Suitable barrier materials also include, but are not limited to, opaque metals, opaque ceramics, opaque polymers, and opaque cermets, and combinations thereof.

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Opaque cermets include, but are not limited to, zirconium nitride, titanium nitride, hafnium nitride, tantalum nitride, niobium nitride, tungsten disilicide, titanium diboride, and zirconium diboride, and combinations thereof.

The barrier layers may be deposited by any suitable process including, but not limited to, conventional vacuum processes such as sputtering, evaporation, sublimation, chemical vapor deposition (CVD), plasma enhanced chemical vapor deposition (PECVD), electron cyclotron resonance-plasma enhanced vapor deposition (ECR-PECVD), and combinations thereof.

The decoupling layer can be produced by a number of known processes which provide improved surface planarity, including both atmospheric processes and vacuum processes. The decoupling layer may be formed by depositing a layer of liquid and subsequently processing the layer of liquid into a solid film. Depositing the decoupling layer as a liquid allows the liquid to flow over the defects in the substrate or previous layer, filling in low areas, and covering up high points, providing a surface with significantly improved planarity. When the decoupling layer is processed into a solid film, the improved surface planarity is retained. Suitable processes for depositing a layer of liquid material and processing it into a solid film include, but are not limited to, vacuum processes such as those described in United States Patent Nos. 5,260,095, 5,395,644, 5,547,508, 5,691,615, 5,902,641, 5,440,446, and 5,725,909, which are incorporated herein by reference, and atmospheric processes such as spin coating and/or spraying.

One way to make a decoupling layer involves depositing a polymer precursor, such as a (meth)acrylate containing polymer precursor, and then polymerizing it *in situ* to form the decoupling layer. As used herein, the term polymer precursor means a material which can be

polymerized to form a polymer, including, but not limited to, monomers, oligomers, and resins. As another example of a method of making a decoupling layer, a preceramic precursor could be deposited as a liquid by spin coating and then converted to a solid layer. Full thermal conversion is possible for a film of this type directly on a glass or oxide coated substrate. Although it cannot be fully converted to a ceramic at temperatures compatible with some flexible substrates, partial conversion to a cross-lined network structure would be satisfactory. Electron beam techniques could be used to crosslink and/or densify some of these types of polymers and can be combined with thermal techniques to overcome some of the substrate thermal limitations, provided the substrate can handle the electron beam exposure. Another example of making a decoupling layer involves depositing a material, such as a polymer precursor, as a liquid at a temperature above its melting point and subsequently freezing it in place.

One method of making the composite of the present invention includes providing a substrate, and depositing a barrier layer adjacent to the substrate at a barrier deposition station. The substrate with the barrier layer is moved to a decoupling material deposition station. A mask is provided with an opening which limits the deposition of the decoupling layer to an area which is smaller than, and contained within, the area covered by the barrier layer. The first layer deposited could be either the barrier layer or the decoupling layer, depending on the design of the composite.

In order to encapsulate multiple small environmentally sensitive devices contained on a single large motherglass, the decoupling material may be deposited through multiple openings in a single shadow mask, or through multiple shadow masks. This allows the motherglass to be

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subsequently diced into individual environmentally sensitive devices, each of which is edge sealed.

For example, the mask may be in the form of a rectangle with the center removed (like a picture frame). The decoupling material is then deposited through the opening in the mask. The layer of decoupling material formed in this way will cover an area less than the area covered by the layer of barrier material. This type of mask can be used in either a batch process or a roll coating process operated in a step and repeat mode. With these processes, all four edges of the decoupling layer will be sealed by the barrier material when a second barrier layer which has an area greater than the area of the decoupling layer is deposited over the decoupling layer.

The method can also be used in a continuous roll to roll process using a mask having two sides which extend inward over the substrate. The opening is formed between the two sides of the mask which allows continuous deposition of decoupling material. The mask may have transverse connections between the two sides so long as they are not in the deposition area for the decoupling layer. The mask is positioned laterally and at a distance from the substrate so as to cause the decoupling material to be deposited over an area less than that of the barrier layer. In this arrangement, the lateral edges of the decoupling layer are sealed by the barrier layer.

The substrate can then be moved to a barrier deposition station (either the original barrier deposition station or a second one), and a second layer of barrier material deposited on the decoupling layer. Since the area covered by the first barrier layer is greater than the area of the decoupling layer, the decoupling layer is sealed between the two barrier layers. These deposition steps can be repeated if necessary until sufficient barrier material is deposited for the particular

application.

When one of the barrier stacks includes two or more decoupling layers, the substrate can be passed by one or more decoupling material deposition stations one or more times before being moved to the barrier deposition station. The decoupling layers can be made from the same decoupling material or different decoupling material. The decoupling layers can be deposited using the same process or using different processes.

Similarly, one or more barrier stacks can include two or more barrier layers. The barrier layers can be formed by passing the substrate (either before or after the decoupling layers have been deposited) past one or more barrier deposition stations one or more times, building up the number of layers desired. The layers can be made of the same or different barrier material, and they can be deposited using the same or different processes.

In another embodiment, the method involves providing a substrate and depositing a layer of barrier material on the surface of the substrate at a barrier deposition station. The substrate with the barrier layer is moved to a decoupling material deposition station where a layer of decoupling material is deposited over substantially the whole surface of the barrier layer. A solid mask is then placed over the substrate with the barrier layer and the decoupling layer. The mask protects the central area of the surface, which would include the areas covered by the active environmentally sensitive devices. A reactive plasma can be used to etch away the edges of the layer of decoupling material outside the mask, which results in the layer of etched decoupling material covering an area less than the area covered by the layer of barrier material. Suitable reactive plasmas include, but are not limited to, O_2, CF_4, and H_2, and combinations thereof. A

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layer of barrier material covering an area greater than that covered by the etched decoupling layer can then be deposited, sealing the etched decoupling layer between the layers of barrier material.

To ensure good coverage of the edge of the decoupling layer by the barrier layer, techniques for masking and etching the decoupling layer to produce a feathered edge, i.e., a gradual slope instead of a sharp step, may be employed. Several such techniques are known to those in the art, including, but not limited to, standing off the mask a short distance above a polymer surface to be etched.

The deposition and etching steps can be repeated until sufficient barrier material is deposited. This method can be used in a batch process or in a roll coating process operated in a step and repeat mode. In these processes, all four edges of the decoupling layer may be etched. This method can also be used in continuous roll to roll processes. In this case, only the edges of the decoupling material in the direction of the process are etched.

If a composite is made using a continuous process and the edged sealed composite is cut in the transverse direction, the cut edges will expose the edges of the decoupling layers. These cut edges may require additional sealing if the exposure compromises barrier performance.

One method for sealing edges which are to be cut involves depositing a ridge on the substrate before depositing the barrier stack. The ridge interferes with the deposition of the decoupling layer so that the area of barrier material is greater than the area of decoupling material and the decoupling layer is sealed by the barrier layer within the area of barrier material. The ridge should be fairly pointed, for example, triangular shaped, in order to interrupt the deposition and allow the layers of barrier material to extend beyond the layers of decoupling material. The

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ridge can be deposited anywhere that a cut will need to be made, such as around individual environmentally sensitive devices. The ridge can be made of any suitable material, including, but not limited to, photoresist and barrier materials, such as described previously.

While certain representative embodiments and details have been shown for purposes of illustrating the invention, it will be apparent to those skilled in the art that various changes in the compositions and methods disclosed herein may be made without departing from the scope of the invention, which is defined in the appended claims.

What is claimed is:

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